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A CASE STUDY OF THE DEVELOPMENT OF A MILITARY
SPECIFICATION - ZIRCONIUM P. (U) ARMY LAB COMMAND
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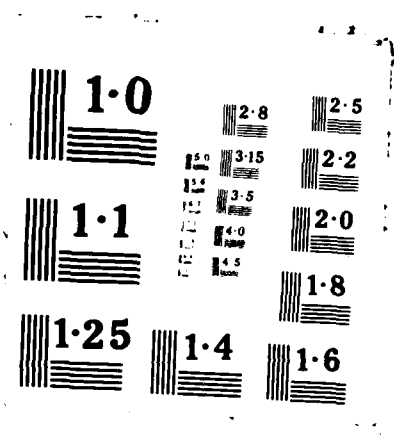
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A CASE STUDY OF THE DEVELOPMENT OF A MILITARY SPECIFICATION — ZIRCONIUM POWDER FOR THERMAL BATTERIES

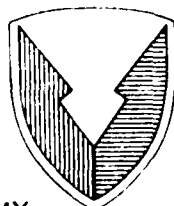
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ABSTRACT

Thermal batteries are used as the critical power source in nuclear fuses, missiles and aircraft. Heat sources in thermal batteries use a fine zirconium powder. This report focuses on the zirconium problem and the development of a military specification for zirconium powder as produced by the magnesium reduction method.

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INTRODUCTION

Thermal batteries are used as the critical or principal power source in almost all U.S. nuclear fuses, missiles and aircraft because of their high reliability, long shelf life and wide temperature range. Thermal batteries derive their name from the fact that their activation depends on the melting of a solid electrolyte by a pyrotechnic source. In a typical cell (see Figure 1), the electrolyte consists of a potassium chloride/lithium chloride eutectic mixture, a calcium metal anode, a CaCrO_4 cathode (depolarizer), and a pyrophoric heat mixture of Zr and BaCrO_4 . A very small current from an electrical match can be used to ignite this pyrophoric mixture. This mixture burns in a controlled manner without releasing a large amount of gas, allowing the term "gasless" to describe the heat source.

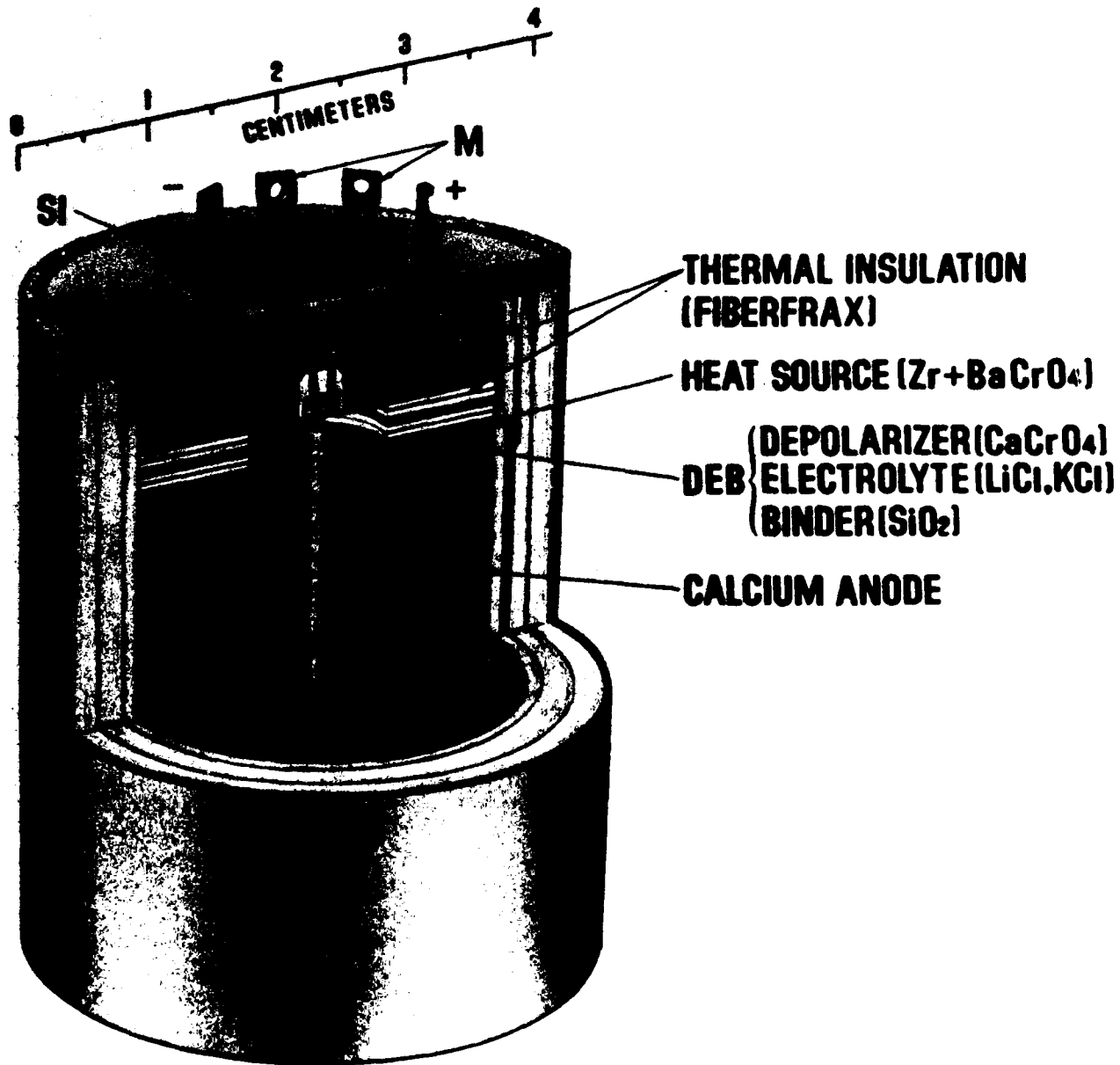


Figure 1. Power source for fusing and guidance systems in missiles.

Heat sources in thermal batteries, fuses and ignites are important pyrophoric applications that use zirconium as fine grain powder ($10\text{ }\mu\text{m}$). Larger grain materials have been studied in solid rocket propellants.

The zirconium metal powder is produced by the reduction of ZrO_2 , which is manufactured from zircon (ZrSiO_4) raw material. In 1972-73, the demand for Australian zircon sand exceeded the supply; this brought about a ten-fold increase in cost of the material and a search for domestic sources. In 1972, the only domestic source (Foote Mineral Company) of calcium reduced fine-grained Zr ceased production after a devastating fire in their zirconium production facility, forcing the thermal battery industry to use magnesium reduced material. Specifications for this material were not available as they were for the calcium reduced material.

Zirconium powder with the required properties became so short in supply that a Joint Deputies for Laboratories Committee Subpanel (JDLC) was chartered in 1975 to investigate the problems. A study was initiated at AMMRC to create a specification for magnesium reduced material to be used in quantitative physical characterization procedures for these new powders. The specific objective of the zirconium powder characterization program established a definitive specification for the purchase of zirconium metal powder used in thermal battery heat mixtures and criteria for monitoring its production.

At this same time (1974-75), routine surveillance discharge tests of stored cup-and-cover design thermal batteries from lots of batteries identical to those installed in DoD systems indicated a high statistical probability of unacceptable performance of such batteries. The AMMRC material characterization study of dissected, stored, unactivated thermal battery components from suspect lots showed that failure was due to degradation of the calcium anode during storage. Further study on thermal batteries established baseline data on material components prior to long-term storage.

The thermal batteries examined in the study are shown in Figure 2 (TN T9-3), and are described as pellet-type batteries. They include: Post separation activation (PSA), arming and fusing (AFS), pulse, and impact detector (ID).

Routine non-destructive tests were carried out on the batteries, followed by dissection of the unactivated batteries. Manufacturers' quality control and deterioration or "aging" of components were critically evaluated as the origins of unacceptable performance.

During this period of time, there were several problems impacting on thermal batteries. This report focuses on the zirconium problem and the development of a military specification to help alleviate it. The investigation of zirconium powder focused on statistically meaningful particle size distribution measurements, precise multiple point surface area and permeability analyses (Fisher Subsieve Size). The powders analyzed in the AMMRC study were supplied by the Ventron Co., which was the only supplier of zirconium powder for thermal battery heat paper mixtures after Foote Mineral ceased production of zirconium powder. The study consisted of a random set of Type I and Type II zirconium powders, where Type I powder was a large grain material of nominal size range equal to $2.0 - 3.0\text{ }\mu\text{m}$, and Type II was a finer grain powder varying from $1.5 - 2.0\text{ }\mu\text{m}$.

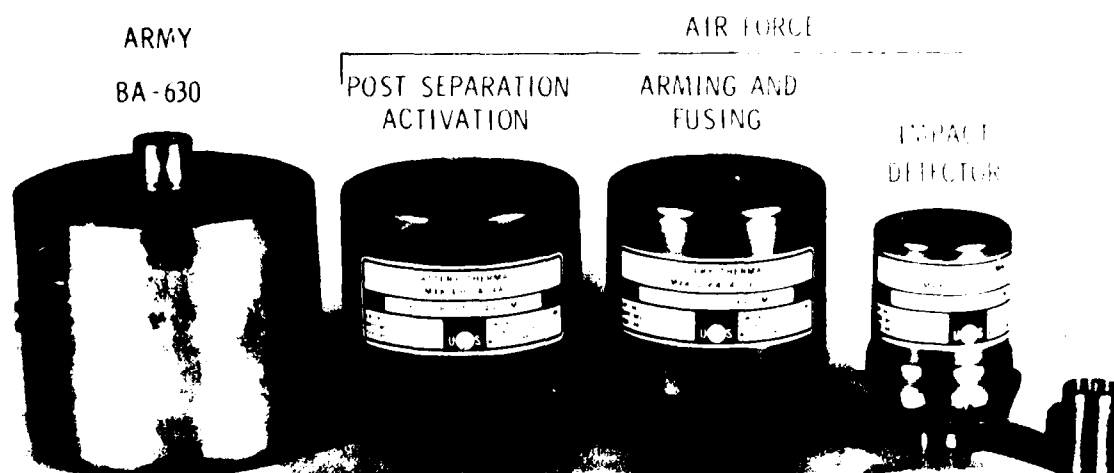


Figure 2. Various types of thermal batteries

SPECIFICATION DEVELOPMENT

In compliance with its mission, the Advanced Materials Standardization Branch of Engineering Standardization Development at the U.S. Army Materials Technology Laboratory (MTL) produced a specification covering zirconium powder using technical information obtained by its Characterization Division.

This action fulfills a general policy important to military logistics. Standardization by the Military establishes requirements to be met by contractors. The mission of the DoD with respect to standardization is to develop, establish and maintain a comprehensive and integrated system of technical documentation in support of designed development, engineering, acquisition, manufacturing, maintenance and supply management. These considerations contribute to the improvement of efficiency and effectiveness of logistical support and operational readiness of the Military Services. It also conserves money, manpower, time, production, facilities and material resources.

The evaluation and justification for the development of all new or major revisions to standardization documents by the preparing activity must include a number of considerations related to cost, improved technical information, consolidation or elimination of existing DoD or non-Government specifications, and industry input.

The preparing activity or military coordinating activity is responsible for managing standardization document projects in accordance with the project system established. Review activities and custodians of other military departments cooperate with the preparing activity on such projects. The preparing activity also has

the continuing responsibility for maintaining military documents current with the state-of-the-art and the requirements of the military departments.

The military standardization documents are coordinated by the preparing activity with representatives of industry as well as interested areas in the Department of Defense. Industry comments are solicited to obtain information on their technical content.

Standardization documents are a vital element in the acquisition of most commodities by the DoD. Standardization documents must contain valid requirements that reflect the user's needs, current technology, modern industrial practices and relevant testing and packaging methods. The standardization document is maintained as a continuing responsibility from publication to cancellation by the preparing activity.

Based on the above description of the Defense Standardization (DoD 4120.3M), specifications develop as a result of the continued effort of technical experts from many sources. A specification such as "Zirconium Used in Gasless Mixtures" took a number of years from the initial research and development program with active MTL/DoD and industry participation.

ZIRCONIUM METALLURGY

Zirconium was discovered as a chemical element by Klaproth in 1789 while analyzing jargon from Sri Lanka. In 1797, Vauquelin gave it the name zirconium and published the properties and preparation of its compounds. Berzelius isolated it in 1824 by heating potassium and potassium hexafluorozirconate in a closed pot. In 1914, the first pure zirconium was prepared by the reduction of zirconium tetrachloride with sodium in a bomb.

The material was produced in 1925 by van Arkel and de Boer, who vaporized zirconium tetraiodide into a bulb containing hot tungsten filament. The tetraiodide was dissociated, and pure zirconium was deposited on the filament. Since hafnium occurs naturally in ores containing zirconium, the "Iodide Crystal Bar" process is still a method of purifying zirconium and hafnium. Because of the similarity in the chemical nature of the two elements, hafnium was not even discovered until 1922. In the late 1940s, scientists had advanced the technological aspects of these metals and became aware of their extremely useful purpose in the nuclear industry. Today, nuclear power dominates the zirconium market, since it is an essential part of the fuel assembly.

In 1947, William J. Kroll developed the commercial reduction technique to produce a pure, ductile zirconium metal by magnesium reduction of zirconium tetrachloride vapor in an inert gas atmosphere. His process was developed at the Bureau of Mines Station in Albany, Oregon. These workers developed the melting and working techniques needed for plate, bars and wire.

Finely divided zirconium powder is made by bomb reduction of zirconium oxide with magnesium or calcium. Because of its large surface area, it is extremely pyrophoric, making it hazardous to produce.

Work performed by Kaufman and Utermeyer showed that early measurements of thermal neutron cross-sections of zirconium were in error due to the presence of

hafnium, which occurs naturally with zirconium in these ores. When the hafnium was removed, zirconium was found to have a very low thermal neutron capture cross-section (rate of absorption of neutrons), making it one of the most transparent metals. This relative transparency to thermal neutrons, coupled with excellent corrosion resistance and good mechanical properties, made zirconium the ideal metal for cladding the atomic fuel in reactors planned for the nuclear submarine program.

The method for reducing the hafnium content to a few hundred parts per million was developed at Oak Ridge. As a result of the efforts of Admiral Hyman Rickover of the U.S. Navy Nuclear Propulsion Program, the properties of zirconium and hafnium provided the essential initial incentive for the technological development in the study and application of nuclear power reactors.

In the late 1950s to the mid 1960s, the chemical process industry began using zirconium in severe corrosion environments, the first commercial power reactors. By 1965, stainless steel was replaced by zirconium alloys, and these alloys became the predominant cladding material for water-cooled reactors. This resulted in a substantial reduction in fuel cost. Much of this technology was contributed through the efforts of investigators in the laboratories of Atomic Energy of Canada, Ltd.

Zircon has been a gem mineral since Biblical times; it was known as jargon in Ceylon and hyacinth in France. The name probably came from the Arabic jargon, relating to the gold or dark amber color of the more common gemstone. Zirconium occurs naturally as a silicate in zircon and other oxide compounds. A common source of zirconium is the mineral zircon, zirconium orthosilicate. Zircon can be found distributed in the earth's crust in such familiar forms as granular limestone, granite, sandstone and other minerals. It is found worldwide; e.g., Kerala State (India), Sri Lanka, Australia, Florida and the Republic of South Africa. All commercial sources of zircon are derived from the mining of deposits due to weathering and concentration of heavy mineral sands in river beds and ocean beaches. The deposits from these sources are collected and wet concentrated to produce a heavy mineral concentrate. The concentrate is dried and separated by various gravity, magnetic and electrostatic separations to yield individual concentrates, including zircon. Other sources of naturally occurring zirconium oxides have been found in South America, Africa and Greenland.

Zirconium is resistant to corrosion by water and steam, mineral acids, strong alkalis, organic acids, salt solutions and molten salts. This property is attributed to the presence of a dense adherent oxide film that forms at ambient temperatures.

Nuclear power provides a stable base for the industry. Industrial uses will increase as zirconium is used in place of other materials to raise productivity or reduce operating costs.

Zirconium hydride in powder form was produced by the reduction of zirconium oxide with calcium hydride in a bomb reactor. Many fires and explosions occurred when the calcium oxide was dissolved with hydrochloric acid to recover the hydride powder. Since zirconium metal is readily available via the Kroll process, zirconium hydride can be produced by the exothermic absorption of hydrogen by pure zirconium. Above 40 wt% hydrogen content, zirconium hydride is brittle at room temperature. This behavior and the reversibility of the hydride reaction are utilized in preparing zirconium alloy powders for powder metallurgy purposes by the hydride-dehydride process.

Zirconium hydrides react with unsaturated organic molecules, a process called hydrozirconation that replaces the hydrogen with the unsaturated group. A stable zirconium compound for this reaction would be cyclopentadienyl zirconium hydrochloride.

THERMAL BATTERY HISTORY

Nuclear power is the greatest market for zirconium. The property of zirconium that makes it desirable for use in nuclear power reactors is its transparency to the passage of thermal neutrons that cause fission of other uranium atoms with the evolution of heat. The electrochemical power sources used in nuclear weapons have been lead-acid, nickel-cadmium, silver-zinc, and thermal batteries. The aqueous electrolyte batteries all had a critical deficiency; their wet stand time was too short. The thermally activated battery solved the wet stand problem. The thermal battery contains a pyrotechnic heat source that ignites and thermally activates the battery, thus melting the electrolyte. The properties of the electrolyte provide long shelf life and good electrical performance of the thermal battery.

During World War II, Dr. George Erb, working in Germany, developed the first practical cells for the thermal battery, using a salt mixture as an electrolyte. The information compiled by Dr. Erb and his co-workers was conveyed to the Americans at the U.S. Ordnance Development Division of the National Bureau of Standards, later to be known as the Harry Diamond Laboratories. The capabilities of thermal cells for ordnance applications was demonstrated. This information led to the development of gasless pyrotechnics and then to the development of the self-contained thermal battery.

The early thermal battery designs had three distinguishing characteristics; heat-paper pads, electrolyte and depolarizer pads, and intercell electrical connectors. The later pellet-type batteries used heat powder pellets, electrolyte/depolarizer pellets and no intercell connection.

ZIRCONIUM SPECIFICATION

As a result of extensive in-house efforts to review the work of industrial and government personnel in the thermal battery field, the recommendation for a zirconium powder specification was presented to the Standardization Division in 1981. The initial stage in the program was the preparation of a Purchase Description for Zirconium Used in Gasless Mixtures, which described zirconium powder applied to zirconium produced by the magnesium reduction process. It covered zirconium metal in fine powder form for use in the manufacture of rapid-rise-time thermal batteries AMMRC-PD-106 (12 July 1982). An earlier purchase description for zirconium powder was TL-PD-105 (21 December 1954).

The early source for zirconium powder, Foote Mineral, was no longer available; after a fire in 1972, the company discontinued the business. The next and present source of availability for zirconium was Morton Thiokol (Ventron Division). While the prevailing Ventron material met the requirements, a study was undertaken to determine whether it was satisfactory for use in thermal batteries being built for the Army. Initial studies with Ventron Type I and Type II zirconium powder revealed that it was different from the zirconium produced by Foote Mineral. Type I zirconium

was determined to be larger particle size and Type II smaller than the Foote Mineral zirconium.

Burning time of zirconium powders is related to the surface area. Both types of Ventron material burn differently than the Foote Mineral material. At Ventron, zirconium is manufactured by the magnesium reduction process, while Foote Mineral used a calcium reduction process that produced a powder of high burning speed. Some of these problems have been solved by "in-house" processes.

Experience with zirconium powder with an average particle size (APS) less than 2.0 microns is hazardous to handle due to spontaneous ignition from static electricity or heat. Ventron Type II zirconium powder (APS 1.5 - 2.0 microns) produced incomplete reduction and yielded a powder of great sensitivity to ignition. This presented serious safety problems for the users at Eagle-Picher.

Despite the fact that both companies claimed to purchase their oxides from the same supplier, Ventron's zirconium was smaller in size and not as reproducible from lot to lot. The problem appears to be due to the fact that Ventron proposed a "broader" specification for zirconium powder than Eagle-Picher's purchase specification. This posed a 'efinite need to study and evaluate the various parameters of zirconium oxide and zirconium to determine their effect on the finished products.

A Thermal Battery Subpanel subgroup was formed to study Toxic/Critical Materials. A questionnaire was prepared and sent to thermal battery vendors to determine the criticality status of their materials. This situation resulted from the summary report concerning chemicals essential for thermal battery production (1974). A DoD directive was written in 1976 to establish policies and assign responsibilities within each DoD component to assure that timely action was initiated when essential end item production capabilities were endangered by the actual or impending loss of manufacturing sources or by material shortages.

A thermal battery standardization and quality control meeting was held at AMMRC in 1978 to review the JDLG Thermal Battery Subpanel Activities Overview. After this meeting, a summary of the improved characterization techniques for thermal battery materials was written which described the breakthroughs accomplished at AMMRC (Wong, Demarco, and McCauley, October 1978). A proposed draft of the purchase description for zirconium powders used in gasless heat powder mixtures was written based on the Ventron practice.

A purchase description was finally prepared as AMMRC-PD-106 (12 July 1982) - Zirconium Used in Gasless Mixtures. This purchase description was converted into the Military Specification, MIL-Z-46189 (2 October 1986). Four drafts of the proposed specification were written and coordinated with interested areas in the DoD and members of industry working with zirconium. The specification for zirconium powder applies to zirconium produced by the magnesium reduction process and covers zirconium metal in fine powder form for use in the manufacture of thermal batteries in most DoD missile systems.

Technical advice was contributed by N. Corbin and T. Resetar in the area of particle size distribution. Personnel at Morton Thiokol were able to add their "hands on" expertise to the development of the specification. A meeting was held at MTL (14 November 1985) in which differences of opinion relating to the two types

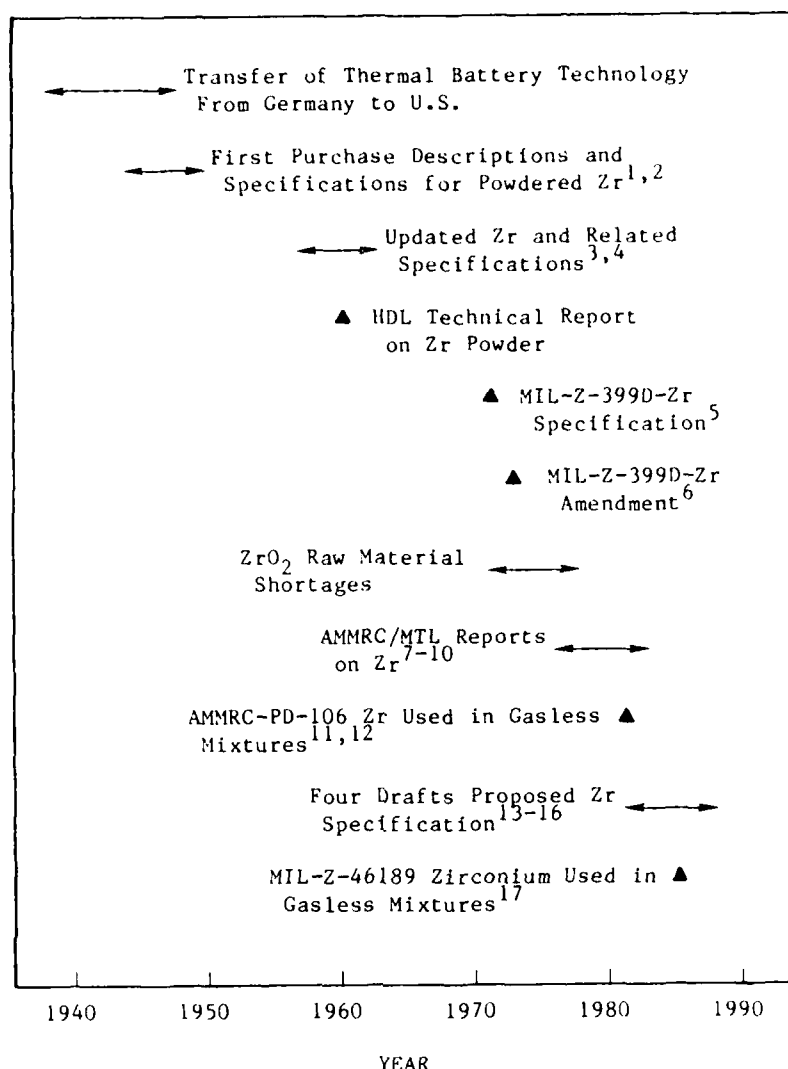
of zirconium were discussed. As a result of this meeting, fine points were clarified and the final draft in the preparation of the military specification was, as noted earlier, published on 2 October 1986.

It is important to note that the preparation of a complex military specification involves much time and cooperation from many sources including military, federal, industrial and academic centers. As shown in Figure 3, the time period for the development of MIL-Z-46189 took over forty years from the initial work contributed by Harry Diamond Labs to the finalization of the military specification as noted above.

The final recommendations for this zirconium powder specification were the result of extensive in-house effort and reviews from government and non-government personnel in the thermal battery field.

ACKNOWLEDGMENTS

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Notes

- 1 Jan Z 399 Zirconium, Powdered (For Use in Ammunition), 25 September 1946
- 2 MIL Z 399A Zirconium (Granular and Powdered), 13 May 1948
- 3 TL PD 105 Purchase Description for Zirconium Powder, 21 December 1959 (Amendment No. 4, 4 June 1964)
- 4 TL PD 129 Purchase Description for Heat Powder Slurry, 2 May 1961
- 5 MIL Z 399D Zirconium (Granular and Powdered), 1 October 1971
- 6 MIL Z 399D Amendment No. 1, 5 April 1973
- 7 J.W. McCauley et al. Annual Summary X.O. 48502, Revision of MIL Z 399D, FY 78
- 8 P. Wong and J.W. McCauley Annual Summary X.O. 38604, Revision of Techniques for Thermal Battery Materials, FY 78
- 9 P. Wong and J.W. McCauley Standardization Meeting, AMMRC, 16 June 1978
- 10 Final Recommendation for Zirconium Powder Specification, 14 December 1981
- 11 AMMRC PD 106 Zirconium Used in Gasless Mixtures, 12 July 1982
- 12 Proposal to Convert AMMRC PD 106 Zirconium Used in Gasless Mixtures to a Military Specification, January 1983
- 13 Draft No. 1 Proposed Military Specification Zirconium Used in Gasless Mixtures, MIL Z XXXXX, 25 April 1983
- 14 Draft No. 2 Proposed Military Specification Zirconium Used in Gasless Mixtures, MIL Z XXXXX, 1 October 1983
- 15 Draft No. 3 Proposed Military Specification Zirconium Used in Gasless Mixtures, MIL Z XXXXX, 28 February 1985
- 16 Draft No. 4 Proposed Military Specification Zirconium Used in Gasless Mixtures, MIL Z XXXXX, 25 June 1986
- 17 MIL Z 46189 Zirconium Used in Gasless Mixtures, 20 October 1986

Figure 3. Sequence of events leading to the development of MIL-Z-46189 - "Zirconium Used in Gasless Mixtures".

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